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The effect of thermoplastic processing on the structure and mechanical properties of metastable austenitic steel

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Abstract. The patterns of structural transformations and the change in the mechanical properties of a metastable austenitic steel subjected to cold plastic deformation by drawing with high total degrees of compression and subsequent aging were studied in this work. The studies showed that after quenching, the use of intense plastic deformation by drawing revealed the high technological capabilities of the studied austenitic steel. Austenite in the steel is deformation-unstable and turns into deformation martensite during cold plastic deformation by drawing. Aging of deformed steel causes an additional increase in mechanical properties, due to the separation of the intermetallic phase NiAl from the bcc solid solution (strain martensite). It was revealed that almost carbon-free corrosion-resistant austenitic steel, as a result of correctly selected alloying, combines the advantages of three steels: metastable austenitic steels, Transformation-Induced Plasticity (TRIP)-steels and martensitic aging steels. As a result of using all possible hardening mechanisms, a high-strength state was achieved.

1. Introduction

The use of high-strength, corrosion-resistant steels makes it possible to obtain products with high service properties, to reduce metal loss, and to reduce the metal consumption of products. Martensitic, austenitic and austenitic-ferritic grades are used for the production of corrosion-resistant wire, which is employed in instrumentation, mechanical engineering, and the manufacture of medical instruments. The specific working conditions of most elastic components, as well as the core medical instrument, require the use of steels and alloys with a high level of strength, considerable elasticity, sufficient ductility and increased corrosion resistance [1].

The formation of a high-strength state in steels is achieved by choosing the appropriate alloying principles and obtaining the desired structural class of the material, as well as by combining various hardening mechanisms: solid-solution hardening, strain hardening in matrix phases without phase transitions, strain hardening due to $\gamma \rightarrow \alpha$ transformation and dispersion hardening with the release of intermetallic phases. In addition, the creation of a nanocrystalline state in the structure of the material leads to the formation of a fundamentally new complex of high physical and mechanical properties. It has been established [2–4] that nanostructured materials and alloys (i.e., ultrafine-grained materials with grain sizes ≤ 100 nm) exhibit very high hardness, strength, impact strength and wear resistance.

In the framework of this study, a new high-strength, corrosion-resistant steel of the austenitic class was developed. This is a steel in which all possible hardening mechanisms can be realized, and which possesses the nanocrystalline structure necessary for providing high mechanical properties on a wire designed for the manufacture of elastic elements [5].

The aim of this work is to study the structure and mechanical properties of austenitic, corrosion-resistant steel at each technological stage of obtaining high-strength wire: depending on the temperature of heating for quenching, after cold plastic deformation by drawing, and also as a result of post-deformation aging.

2. Research methodology

Corrosion-resistant steel of the austenitic class was selected as the material for study: 03Kh13N10K5M2YuT steel alloying systems 0.03 % C – 13 % Cr – 10 % Ni – 5 % Co – 2 % Mo – 0.8 % Al – 0.4 % Ti. The influence of quenching the heating temperature was studied at a temperature range of 800–1300 °C. Cold plastic deformation was carried out by drawing blanks from a diameter of 14.3 to 2.77 mm at room temperature. Samples were taken along the drawing route to study structural changes during deformations with compression ratios of 30 % (e = 0.39), 41 % (e = 0.52), 69 % (e = 1.15), 80 % (e = 1.60), 88 % (e = 2.17), 94 % (e = 2.32) and 96 % (e = 3.27). After deformation, aging was carried out at a temperature range of 300–700 °C for 1 h.

Mechanical tests were carried out on wire samples; the tensile strength (σ_s), yield strength ($\sigma_{0.2}$), relative narrowing (ψ) and elongation (δ) were determined. X-ray phase analysis was carried out on a DRON-2 diffractometer. The amount of the magnetic phase was determined on a Faraday scale. Hardness was measured using a Vickers instrument. Microhardness was measured on a Leco PC series automatic hardness tester with a programmable pitch and a load of 0.001 kg. Metallographic studies were performed on a Neophot optical microscope. Electron microscopic studies included a study of the fine structure of the steel and the determination of phase composition using a JAM 200-CX microscope.

3. Research results and discussion

In the framework of this work, a new high-strength, corrosion-resistant, austenitic carbon-free steel based on a Fe-Cr-Ni base, additionally alloyed with Co, Mo, Ti and Al, designed for the manufacture of high-strength wire of thin and thin sections, was developed. An optimal mode for producing high-strength wire from this new corrosion-resistant steel has been developed [6]. The optimal technological processes are:

1) obtaining a degree of supersaturation of the solid solution on large cross-section blanks by austenitization, followed by quenching in water. After hardening, an austenitic state with a deformation-unstable structure should be formed, which is the basis for obtaining high plasticity during drawing;
2) the subsequent deformation by drawing with large total degrees of compression, in combination with deformation transformation, should provide maximum fragmentation (defectiveness) of the structure, as well as a nanocrystalline structure, as the basis of a high-strength state;
3) further post-deformation aging should provide the necessary form and shape of reinforcing dispersed particles without a significant change in the phase composition of the matrix.

3.1. Quenching temperature selection

Microstructural studies of the steel after quenching from temperatures of 800–1300 °C showed that the steel structure consists of 100 % austenite and contains polyhedral grains with a large number of annealing twins (Figure 1). The higher the temperature, the larger the austenite grain is. Phase X-ray diffraction analysis confirms that after quenching from all the temperatures, the main phase is austenite; however, with an increase in the heating temperature to 1200 °C and higher, a bcc phase (δ-ferrite) appears.

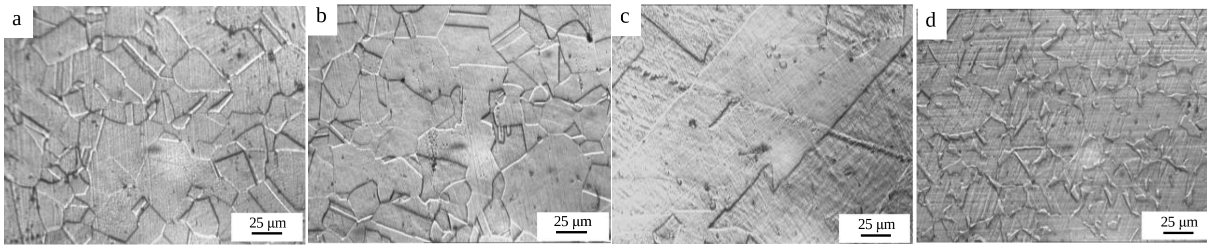


Figure 1. Microstructure of austenitic 03Kh13N10K5M2YuT steel after quenching from various temperatures: (a) 800 °C; (b) 1000 °C; (c) 1200 °C; (d) 1300 °C.

Increased strength after low-temperature quenching is associated with the presence in the γ-solid solution of a high-temperature intermetallic Ni_3Al type phase (Figure 2). The dissolution of this phase with an increase in the heating temperature for quenching to 1000–1100 °C leads to the formation of a homogeneous γ-solid solution, while strength decreases and ductility increases. A further increase in temperature to 1300 °C leads to an increase in hardness due to the appearance of a certain amount of δ-ferrite in the steel structure (see Figure 1, d) as a result of the $\gamma \rightarrow \delta$ transformation.

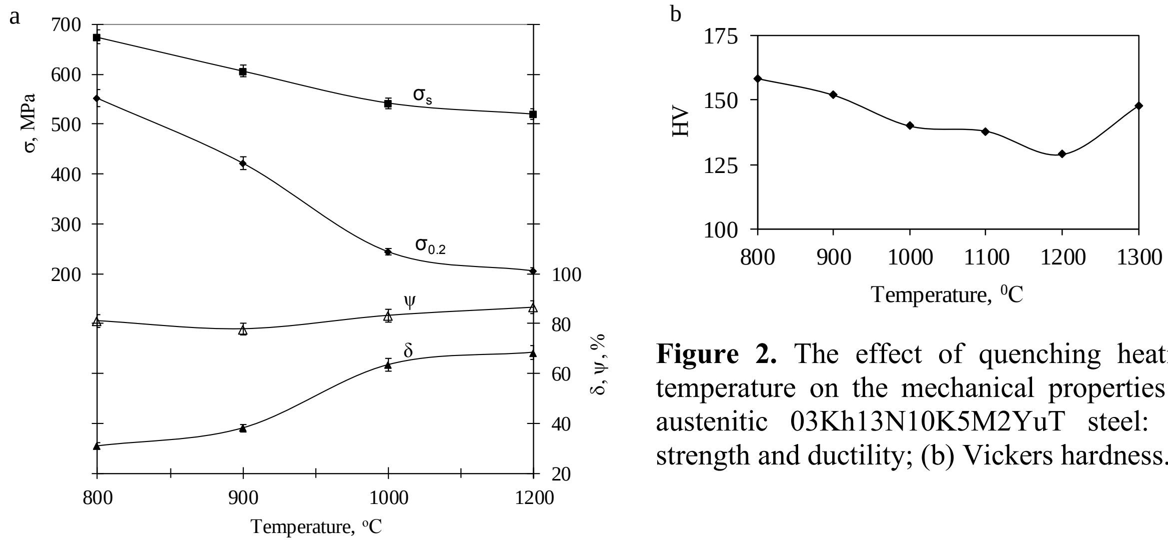


Figure 2. The effect of quenching heating temperature on the mechanical properties of austenitic 03Kh13N10K5M2YuT steel: (a) strength and ductility; (b) Vickers hardness.

Thus, from the point of view of choosing a temperature for quenching for further plastic deformation, the best is quenching from a temperature range of 1000–1100 °C in water. This is due, on the one hand, to the dissolution of excess intermetallic phases that reduce the aging effect and obtain a uniform austenitic structure and, on the other hand, to the absence of intensive grain growth and the formation of δ-ferrite. The mechanical properties of the studied hardened steel after quenching from 1000 °C are: σ_s = 540 MPa, $\sigma_{0.2}$ = 245 MPa, δ = 63 %, ψ = 83 %, and hardness HV = 143.

3.2. Cold plastic deformation by drawing

Cold plastic deformation by drawing is the second stage of the developed regime for producing high-strength wire from new corrosion-resistant steels. Drawing deformation with large total compression ratios, in combination with deformation transformation, should provide maximum fragmentation (imperfection), as well as a nanocrystalline structure, as the basis for a high-strength state.

An increase in the degree of cold plastic deformation to e = 2.32 made it possible to obtain high strength values (σ_s = 1500 MPa): the increase in strength was 960 MPa (Figure 3, a, b). High hardness values were also obtained (425 HV): the increase in hardness was 280 HV. As shown by the data from phase X-ray diffraction analysis (Figure 3, c), the austenite undergoes a martensitic transformation during cold plastic deformation: with a strain of $e = 2.32$, the amount of martensite is about 90 %.

The intensity of martensite formation was studied both by X-ray diffraction and magnetometric methods. The appearance of a martensite strain was observed at a sufficiently large strain ≈ 41%. With further drawing, the amount of martensite steadily increases.

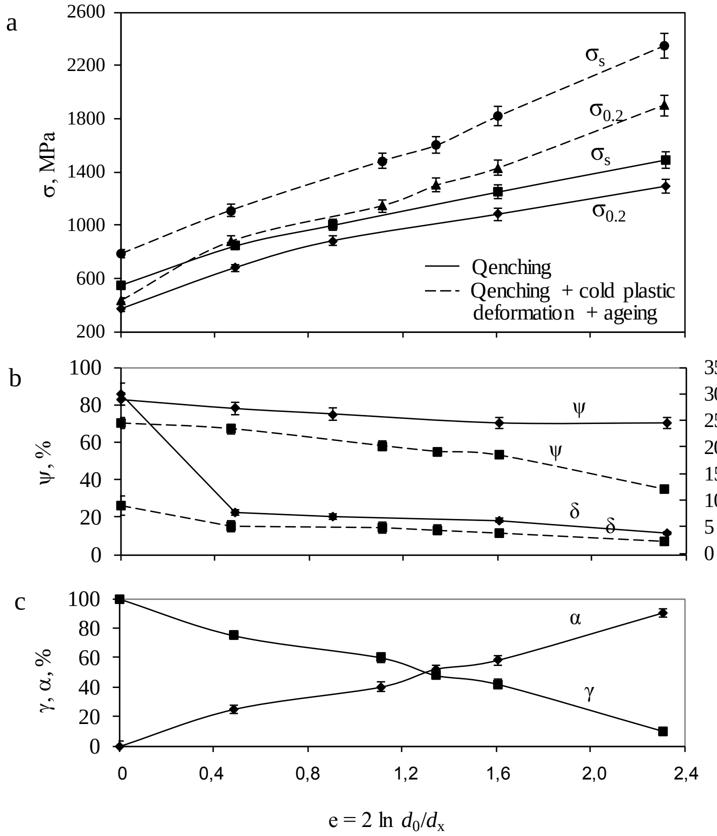


Figure 3. The effect of cold plastic deformation on the properties of hardened austenitic 03Kh13N10K5M2YuT steel: (a) strength properties; (b) plastic properties; (c) phase composition.

To determine the effect of the strain on the steel's structure, microstructural studies of the deformed wire samples were carried out. With moderate compressions (15–30 %), slip bands appear in individual grains. With greater deformation, the shape of the grains' changes: from equiaxed they become more fibrous and elongated along the drawing axis (Figure 4).

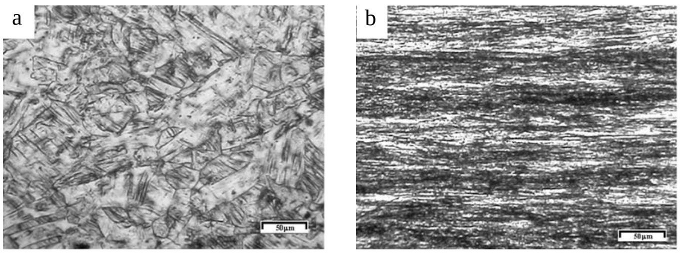
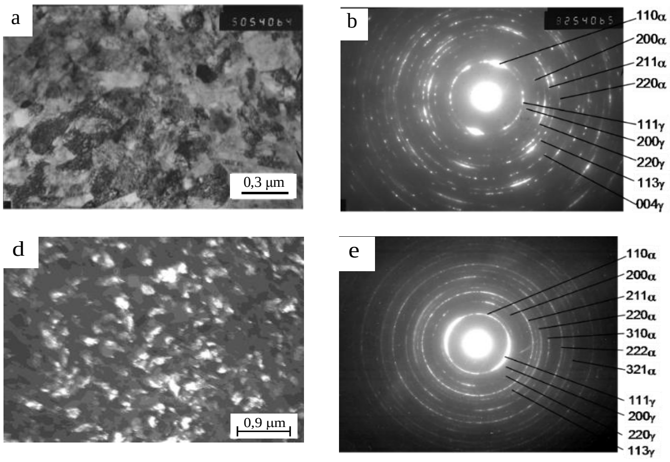


Figure 4. The microstructure of 03Kh13N10K5M2YuT steel after quenching and deformation at e = 0.52 (a) and e = 2.32 (b).

As a result of electron microstructural studies, it was found that, with a total strain of 88 % ($e = 2.17$), ring diffraction patterns appear (Figure 5, b) due to the misorientation of crystals at an angle greater than 5°. This is due to the fine-grained structure with reflections like α and γ phases. With a strain of ≈ 94 % (e = 2.32), martensite crystals with a submicroscopic size ≈100 nm and less occur (Figure 5, e-d).



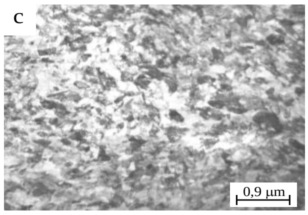


Figure 5. Microstructure of 03Kh13N10K5M2YuT steel after deformation e = 2.17 (ε = 88 %) (a-b) and deformations e = 2.32 (94 %) (c-e): (a), (c) bright field image; (b), (d) microdiffraction; (e) dark-field image in the reflection of the α-phase (110).

Thus, the extremely high ductility found in the steel is due to a martensitic transformation with optimal intensity, which ensures the formation of martensite submicrocrystals. The mechanical properties obtained by stretching the wire after cold plastic deformation e = 2.17 are: σ_s = 1480 MPa, $\sigma_{0.2}$ = 1200 MPa, δ = 4 %, ψ = 70 % and martensite crystal size 100–200 nm. After cold plastic deformation, the mechanical properties are: e = 2.32: σ_s = 1500 MPa, $\sigma_{0.2}$ = 1300 MPa, δ = 3 %, ψ = 70 % and martensite crystals 20–100 nm in size. The extremely high manufacturability of this steel should be noted, as it was able to stretch with very high total deformations without intermediate softening annealing.

3.3. The influence of post-deformation aging

We studied the possibility of further increasing the strength properties, and, in particular, the feasibility of using subsequent aging after quenching and cold plastic deformation. It was revealed that post-deformation aging of the 03Kh13N10K5M2YuT steel at 500 °C for 1 h leads to a significant increase in strength properties (Figure 3, dashed curves). This is a result of aging the bcc phase (strain martensite) with the release of a dispersed ordered intermetallic NiAl phase. The greater the amount of martensite formed in the process of deformation (drawing), the higher the increase in strength properties (σ_s, $\sigma_{0.2}$). Sufficiently high plasticity characteristics (δ = 3 % and ψ = 45 %) are also maintained.

An electron microstructural study of pre-deformed samples of the 03Kh13N10K5M2YuT steel after aging at 500 °C for 1 h showed that the structural components of the deformed and aged steel are the bcc and fcc phases. The main mechanism of aging is the heterogeneous precipitation of the intermetallic phase from the bcc solid solution. In the structure of deformed and aged metastable austenitic steel (Figure 6), a ripple-type contrast is observed in electronic photographs due to the appearance of fine particles. Superstructural reflections belonging to particles of the intermetallic NiAl phase appear on electron diffraction patterns. An increase in temperature and aging time (650 °C, 2 h) leads to an increase in the precipitated particles of the NiAl phase (Figure 6, c).

Thus, the available data allow us to conclude that the presence of martensite is effective for achieving a high-strength state in unstable austenitic steel not only due to hardening from the use of plastic deformation, but also due to subsequent post-deformation aging. At the same time, when analyzing the causes of thermomechanical hardening, one cannot ignore the undoubted influence of the structural state of another solid solution, austenite. Deformation hardening of the latter can also contribute to a general change in the properties of a deformed metal during aging.

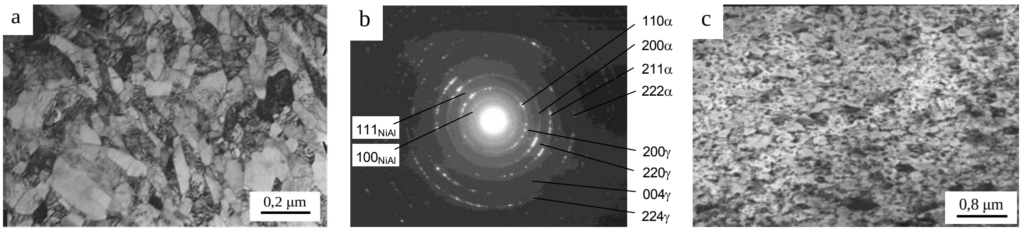


Figure 6. The structure of 03Kh13N10K5M2YuT steel after quenching from 1000 °C, deformation e = 2.32 and aging: at 500 ºC, 1 h (a–b), at 650 ºC, 2 h (c).

4. Conclusions

Cold plastic deformation is a reliable and easy way to achieve a high level of strength. The austenite of the steel under study is deformation-unstable and is almost completely transformed into deformation martensite during cold plastic deformation. The increase in strength reaches 900–1100 MPa (depending on the final diameter) with fairly good ductility. It is shown that the extremely high ductility found in this austenitic steel is due to the occurrence of a martensitic transformation with optimal intensity, which provides for the formation of 20–100 nm martensite submicrocrystals (nanostructures). The aging of deformed steel causes an additional increase in mechanical properties, which is associated with the decomposition processes of a supersaturated bcc solid solution (deformation martensite).

It was revealed that the phase responsible for the hardening of the deformed test steel during aging is an intermetallic NiAl phase. Thus, the available data allow us to conclude that almost carbon-free, corrosion-resistant austenitic steel, as a result of properly selected alloying, combines the advantages of three steels: metastable austenitic steels, TRIP-steels and martensitic aging steels. As a result of using all possible hardening mechanisms, a high-strength state was achieved.

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